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SORPTION CHARACTERISTICS OF ZEOLITE AND BENTONITE NATURAL ADSORBENTS MODIFIED COMPLEX

Abstract. The main objective of our study is to obtain a complex compound from natural adsorbents to purify wastewater with simultaneous sorption of chemical and microbiological substances contained therein, which contribute to disinfection and softening of water, increasing the degree of treated water saturation with calcium, magnesium salts and trace elements, while not requiring the use of sophisticated equipment. The technical result is to create a complex of adsorbents with the sorbing ability of chemical and microbiological pollution, disinfecting and softening water, enriching it with calcium ions, magnesium, sodium, potassium, as well as trace elements.

Key words: water, purification, sorption, natural adsorbents, aluminosilicates, heavy metals.

Introduction. Currently, water purification is becoming one of the most common technological processes in the world, including Kazakhstan. This determines the particular relevance of the issue of reducing the cost of cleaning drinking water, and sewage [1]. In this regard, a very promising seems the application of natural sorbents, deposits of which are available in Kazakhstan. In the literature appears more and more with about the effectiveness of natural sorbents for disperse impurities, heavy metals, oil and oil products, surface active agents, dyes, radioactive contaminants and others [2].

The study was intended to determine the methods of a promising approach, the creation of new types of modified complex sorbents and an experimental evaluation of the effective use of a modified complex of natural mineral sorbents, based on zeolite and bentonite, for the purification and conditioning of drinking water and the purification of wastewater.

Technical characteristics of the studied adsorbents:

Bentonite (Mukrynskoye field of Almaty region) - natural clay mineral, hydroaluminosilicate, has the property of swelling during hydration (14-16 times). In a confined space for free swelling in the presence of water, a dense gel is formed that prevents further penetration of moisture. This property, as well as non-toxicity and chemical resistance, makes it indispensable in industrial production, construction and many other fields of application.

Natural bedding bentonites usually have a pH of 6-9.5 (for 5% aqueous suspension after settling for 1 hour) and contain less than 2% sodium carbonate; the total content of interchangeable sodium and calcium does not exceed 80 me/100. There are two types of bentonites:

- Calcium, with a low degree of swelling;
- sodium, with a high degree of swelling (swelling rate less than 7 ml/g or more than 12 ml/g).

Chemical formula: $AI_2 [Si_4O_{10]} (OH)_2 * nH_2O$.

Chemical composition: SiO_2 - 58.25%; AI_2O_3 - 14.27%; Fe_2O_3 - 4.37%; FeO - 0.5%; Ti_2O - 0.36%; CaO - 2.07%; MgO - 3.67%; P_2O_5 - 0.18%; S - 0.14%; K_2O - 1.2%; Na_2O - 2.25%; PPP - 12.19%.

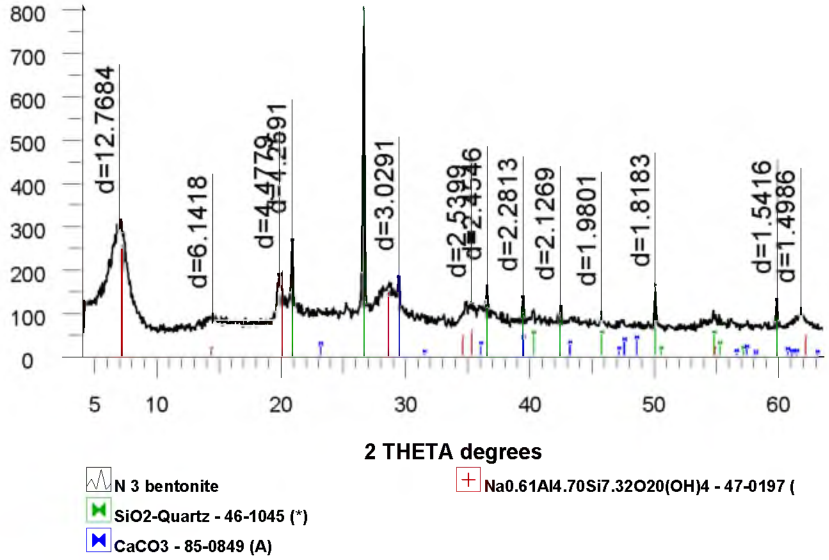


Figure 1 – Radiograph of a sample of bentonite-montmorillonite (Mukrynskoye field of Almaty region)

Zeolites (Maytobinskoye deposit of Almaty region) - a large group of minerals with similar composition and properties; aqueous calcium and sodium aluminosilicates of the subclass of frame silicates, with glass or pearlescent gloss, known for their ability to give and reabsorb water depending on temperature and humidity. The most common representatives of the group of zeolites - natrolit, shabazit, gevlandit, stilbite (desmin), mordenite, thomsonite, lomontite.

The crystal structure of natural zeolites is formed by the tetrahedral groups $SiO_{2/4}$ and $AlO_{2/4}$, united by common vertices in a three-dimensional framework, permeated with cavities and channels (windows) of 2-15 angstroms[3]. The open frame-cavity structure of zeolites [AlSi] O_4^- has a negative charge, compensated by counterions (metal cations, ammonium, alkyl ammonium and other ions, introduced by the mechanism of ion exchange) and easily dehydrating water molecules.

Chemical formula: Zeolite-clinoptilolite, described by the idealized formula $(KNa,)_4\ CaAl_6Si_{30}O_{72} * 24H_2O$ - is a crystalline aqueous aluminosilicate.

Chemical composition: Al_2O_3 - 12.9-13.2%; K_2O - 4.0-4.8%; CaO - 1.8-2.4%; V - 0.001%; Cu - 0.001%; Rb - 0.001%; SiO_2 - 66.2-78.3%; Na_2O - 1.8-2.2%; Fe_2O_3 - 0.8-1.2%; Mn - 0.001%; Be - 0.001%; As - 0.03%.

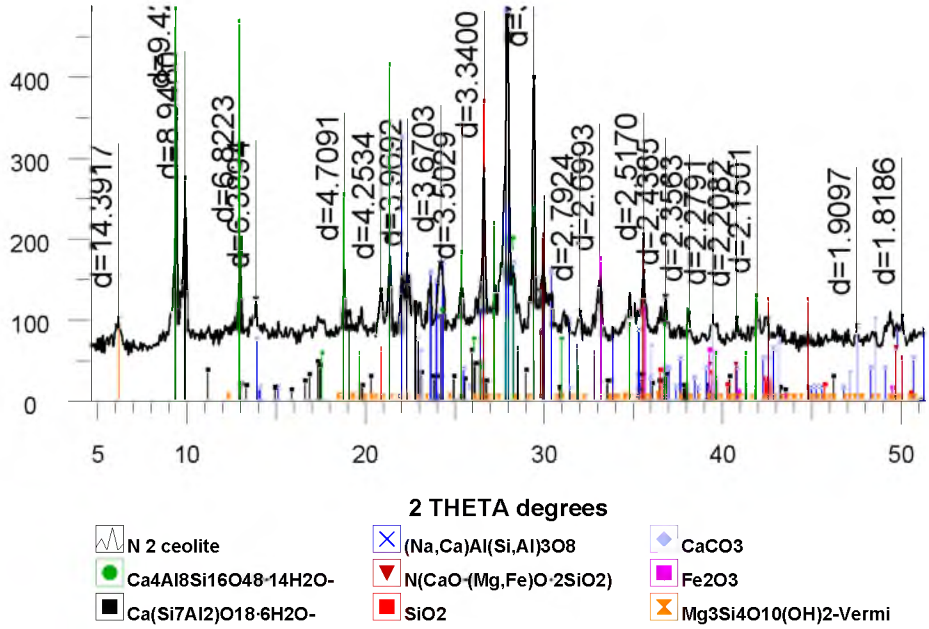


Figure 2 – Zeolite radiograph (mine I Maytobe Almaty region)

Materials and research methods. A sorbent based on zeolites modified by ion exchange of silver ions is known for absorption of radioiodine and/or radiocaesium. After ion-exchange modification, the sorbent is additionally treated with acetylene in a gaseous or liquid medium so that its carbon content in carbon is 0.4 - 2,0 wt. The sorbent is designed specifically for purifying water from strong contamination with radionucleotides, it is effective to purify drinking water, but it does not enrich water with calcium, magnesium salts, as well as with trace amounts of elements from side groups of the Periodic System[4,5].

The technical result is achieved by the fact that the proposed sorbent of the following composition, chemical formula: Zeolite-clinoptilolite, chemical formula: $(KNa)_4\ CaAl_6Si_{30}O_{72}$ * $24H_2O$ is a crystalline aqueous aluminosilicate, chemical composition: AI_2O_3 - 12.9-13.2%; K_2O - 4.0-4.8%; CaO - 1.8-2.4%; V - 0.001%; Cu - 0.001%; Rb - 0.001%; SiO_2 - 66.2-78.3%; Na_2O - 1.8-2.2%; Fe_2O_3 - 0.8-1.2%; Mn - 0.001%; Be - 0.001%; As - 0.03% and Bentonite- hydroaluminosilicate, chemical formula: $AI_2\ [Si_4O_{10}\ (OH)_2$ * $nH_2\ O$, the chemical composition: SiO_2 - 58.25%; AI_2O_3 - 14.27%; Fe_2O_3 - 4.37%; FeO - 0.5%; Ti_2O - 0.36%; CaO - 2.07%; MgO - 3.67%; P_2O_5 - 0.18%; S - 0.14%; K_2O - 1.2%; Na_2O - 2.25%; PPP - 12.19% .

To test the effectiveness of the complex, natural adsorbents were taken in different percentages, agglomerate was obtained: 1) zeolite (60%) and bentonite (40%), 2) zeolite (50%) and bentonite (50%), 3) zeolite (40%) and bentonite (60%). Subsequently, capsules 10 mm wide, 15mm long were made from the mixture obtained, acid activation was performed using 15 % H_2SO_4 taken in an amount of 50 % of the air-dry sample, the duration of treatment was 4 hours. In a muffle furnace at a temperature of 400 degrees, heat treatment was carried out to increase the total porosity.

Determination of the sorbent porosity.

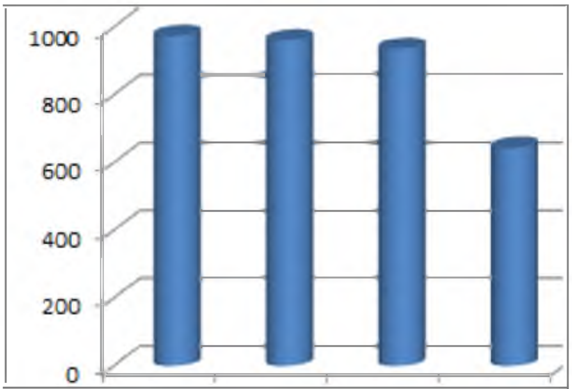
One of the most important characteristics of the adsorbent is the determination of its porosity, which in general depends on its deposit. It is characterized by the total volume of all voids (pores) in the rock [6].

Determination of the porosity of the sorbent was carried out according to the following method. The samples were boiled in a glass with distilled water for 1.5–2.0 hours and then weighed [7].

Sample density, water absorption and porosity were calculated according to table 1, where: m_0 - the mass of the samples with suspension in water, g; m_1 - weight of wet samples, g; m_2 - weight of dry samples g; m_3 - suspension weight year.

Table 1 – Incoming data for calculation (modified complex of bentonite and zeolite)

No experience	Material name	m_0, g	m_1, g	m_2, g	m_3, g
1	Zeolite complex (60%) and bentonite (40%)	10,0018	31.4312	19.0645	0.5923
2	Complex zeolite (50%) and bentonite (50%)	9.6453	29.2576	16.7312	0.4627
3	Zeolite complex (40%) and bentonite (60%)	9,9459	31.2057	18,1124	0.4134



Heavy metals, mg/dm^3	974	961	936	644
	Waste water	Zeolite (60%) Bentonite (40%)	Zeolite (50%) Bentonite (50%)	Zeolite (40%) Bentonite (60%)

The results of the laboratory research on change of heavy metals in wastewater by modified complex of natural adsorbents consisting of bentonite, zeolite in different percentages.

The limits of the relative total error of result, which is admitted, is equal to 2.0; at confidence probability is 0,95. The results of the research are given in table 2.

Table 2 – Determination of porosity of bentonite and zeolite complex

Sample number	The name of material	Sample weight, g	Water adsorption, %	Density, %	Porosity, %
1	Zeolite (60%) and bentonite (40%) complex	21,2145	76.0690	43.8490	73.45
2	Zeolite (50%) and bentonite (50%) complex	20,7124	77.7860	44,7296	73.91
3	Zeolite (40%) and bentonite (60%) complex	19,9437	82,3320	45.6125	80,51

Determination of adsorption capacity according to methyl orange and iodine.

To determine the adsorption according to methyl orange we have chosen the method, introduced at State Standard 4453–74.

For this purpose, the sample of coal was placed in a conical flask with capacity of 100 cm^3, added 25 cm^3 of methyl orange solution. After that, optical density was determined using a photoelectric colorimeter.

As a control solution we used distilled water. From the received optical densities on the base of calibrating graph we determined residual concentration of the pigment.

Adsorption activity was calculated by the formula 1 [5,8]:

$$X = \frac{(C_1 - C_2 K) \cdot 0.025}{m} \tag{1}$$

where C_1 - concentration of the original dye solution, mg/dm^3; C_2 - concentration of the dye solution after interacting with tripoli powder, mg/dm^3; K - dilution factor; m - weight of the coal sample, g; 0.025 - the volume of methyl orange solution, dm^3 .

The results of the research of the adsorption capacity are given in table 3.

Table 3 – The results of the experiment to determine the adsorption capacity according to methyl orange

Sample	Adsorption capacity according to MO			
	Weight of the sample, g	Concentration, mg / dm ^{3}		Adsorption capacity according to MO, mmol / g
		original	residual	
1. Zeolite (60%) and bentonite (40%) complex	0.1	1500	0,600	0.6880
2. Zeolite (50%) and bentonite (50%) complex	0.1	1500	0,800	0.5351
3. Zeolite (40%) and bentonite (60%) complex	0.1	1500	0.750	0.6291

Determination of the adsorption capacity of tripoli by iodine was realized in accordance with the State Standard 4453–74. Iodine number is an approximate measure of the ability of a substance to adsorb small molecules, which depends on the size of the surface area. The processing of the result was carried out according to formula 2 [4,8]:

$$X = \frac{(V_1 - V_2) \cdot 0.0127 \cdot 100 \cdot 1000}{10 \cdot m} \tag{2}$$

where V_1 - the volume of sodium thiosulfate solution (0.1 n), which was used for titration of 10 cm^3 of iodine solution in potassium iodide, cm^3; V_2 - the volume of sodium thiosulfate solution (0.1 n), which was used for titration of 10 cm^3 of iodine solution in potassium iodide, after processing it with bentonite and zeolite complex, cm^3; 0,0127 - the mass of iodine, which corresponds to 1 cm^3 of sodium thiosulfate solution, g; 100 - the volume of iodine solution in potassium iodide, which is needed for bentonite and zeolite complex, cm^3 ; m - the mass of the sample of bentonite and zeolite complex, 1.00g.

The results of calculation of the iodine number of the bentonite and zeolite complex are given in table 4.

Table 4 – The results of the experiment to determine the iodine number of the bentonite and zeolite complex

Sample	Iodine adsorption capacity			
	Weight of the sample, g	The amount of thiosulfate for titration, cm^3		Iodine adsorption capacity, mmol/g
		Iodine	Bentonite and zeolite complex	
1. Zeolite (60%) and bentonite (40%) complex	0.5	16,10	12.60	10.16
2. Zeolite (50%) and bentonite (50%) complex	0.5	16,10	12.40	12.70
3. Zeolite (40%) and bentonite (60%) complex	0.5	16,10	12.52	11.80

Since bacterial spores are much more resistant to disinfecting agents than E.coli cells, the absence of the latter in water does not guarantee absence of the spores. The indicator, showing presence of bacterial spores in water was anaerobic spore-forming organism C. perfringens and aerobic spore-forming bacterium B. subtilis. These bacteria differ in location of spores in a cell. Since their spores are able to exist in water for much longer than coliform bacteria, they are resistant to disinfection and therefore serve as indicators of long-term contamination and defects in filtering techniques at waterworks.

Tables 5-7 show the results of studying the effectiveness of NMS (natural mineral sorbents) regarding removal of E. coli cells from water, as well as B. subtilis and C.perfringens spores.

The results shown in tables 5-7 indicate that NMS effectively remove microbial contamination from water at the concentration of bacterial suspension (1.2÷3.5) $x10^3$ cells/ml.

At increasing the concentration of the bacterial suspension significantly [up to (3.1÷3.2) $x10^4$ cells/ml] in the filtrate inoculations you can find bacteria colonies. At the same time, their number decreases in comparison with the initial by (4÷5)$x10^3$ times - for the filter. The effectiveness of reducing microbial contamination with zeolite is significantly lower than in control, and is (2÷2.8) $x10^3$ times [9].

Table 5 – The number of microorganisms E.coli (X±x) in water before and after filtration through filters (n = 5)

The number of microorganisms, cells/ml			
Initial	Filter type		
	Coal filter	Zeolite filter	Filter- zeolite, bentonite complex.
(1.2 ± 0.1) $x10^3$	0	0	0
(3,5 ± 0,2)) $x10^3$	0	0	0
(3.2 ± 0.2) $x10^3$	(1.5 ± 0.3) $x10^3$	(0.7 ± 0.05) $x10^3$	(0.6 ± 0.03) $x10^3$

Table 6 – The number of microorganisms B. subtilis (X±x) in water before and after filtration through filters (n = 5)

The number of microorganisms, cells / ml		
Initial	Filter type	
	Carbon filter	Filter - zeolite, bentonite complex
(1.5 ± 0.1) $x10^3$	(1.5 ± 0.1) * $x10^1$	(0.7 ± 0.02) $x10^1$
(3.4 ± 0.2) x $x10^3$	(1,8 ± 0.1) * $x10^2$	(1,1 ± 0.02) $x10^2$
(3.2 ± 0.1) $x10^4$	(4,5 ± 0.1) * $x10^2$	(2,3 ± 0.02) $x10^2$

It should be noted that in practice in distribution networks, as well as at water withdrawal from a natural water source, we face less severe microbiological pollution. Installations designed for water disinfection in the field and built on the principle of ultraviolet bactericide irradiation are designed for coli index of not more than 5×10^3 cells/l [10].

Determination of toxicity of water samples containing chemical toxicants (phenol, copper sulphate) before and after passing through NMS filters was performed using Daphnia magna and microalgae Chlorella vulgaris.

The data in table 7 show that water before passing through NMS filters had a toxic effect on the Daphnia magna (the percentage of daphnia death in both cases exceeded 50%). After passing water through NMS filters there was no toxic effect on daphnia in all cases, and the percentage of their deaths hardly differed from the control one, except for the variant with bentonite at concentration of 1.5 mg/l, when the percentage death was 11%.

Table 7 – The effect of NMS on the death of Daphnia magna (% to the control one)
in water samples containing toxicants (n = 5)

Toxicant content mg/l	Original water	Zeolite	Bentonite
10.0 $CuSO_4$	85.5 ± 5.0	4.0 ± 0.2	3.0 ± 0.2
5.0	59.0 ± 3.0	3.0 ± 0.1	3.0 ± 0.2
1.5	73.0 ± 2.5	11.0 ± 0.5	4.0 ± 0.3
0.05	57.0 ± 1.5	3.0 ± 0.2	3.0 ± 0.1

Notes: 1. In the control (cultivation water), no death was noted;

2. The duration of cultivation Daphnia magna in water samples is 96 hours;

3. After passing through the filters, the water pH in all variants of the experiment was in the limits of 7.0 ÷ 7.2. The water was aerated for 1 hour.

Some indicators of the quality of tap water (Taldykorgan city) before and after filtering through filter complex, containing the complex NMS studied in the work (bentonite and zeolite).

Conclusions. The received results testify that after passing tap water through the filter zeolite and bentonite complex the composition of water had significant changes. Its organoleptic characteristics significantly improved, in particular, they began to comply with the Sanitary Regulations and Standards in smell and taste of water.

The iron content in water decreased by 9.5 times and began to comply with the Sanitary Regulations and Standards requirements.

In the filtered water pH, the content of calcium, magnesium, silicon, hydrocarbonate ions, as well as the total hardness and dry residue increased. This fact should be assessed positively, since it is known that tap water is distinguished by low content of these essential elements, increased softness and total low salt content.

Based on the analysis of technical and economic indicators for this experimental study we selected material of the domestic resource base, mineral sorbents of different nature: zeolite and bentonite. Experimentally we selected conditions for NMS activation. Most significantly (by 35-57%) PMS activity increased after acid processing with a mixture of 10% sulfuric acid (1: 1).

The results of the conducted sanitary-microbiological studies have shown that NMS have pronounced sorption properties against bacteria E.coli strain K12, as well as the spores B.subtilis and C.perfringes. All studied NMS and their combination in the filter of zeolite and bentonite complex effectively remove microbial contamination from water at the initial concentration (1.2 ÷ 3.5) x10^3 cells/ml.

Since the work has not been completed yet, it is planned to conduct further studies and to offer improvements of adsorption characteristics of the complex of natural adsorbents for water purification from harmful impurities.

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ЦЕОЛИТ ЖӘНЕ БЕНТОНИТ ТАБИҒИ АДСОРБЕНТТЕРІНЕН ЖАСАЛҒАН МОДИФИКАЦИЯЛАНҒАН КЕШЕННІҢ СОРБЦИЯЛЫҚ СИПАТТАМАЛАРЫ

Аннотация. Мақалада авторлар ауызсу және ластанған сарқынды ақаба суды жоғары дәрежеде тазартуға қол жеткізуге мүмкіндік беретін бентонит, цеолит және диатомит негізіндегі құрамдастырылған табиғи адсорбенттер кешенін алудың тиімді технолоғиясын жасауға тырысты, өйткені зиянды қосылыстармен ластау үрдісі жоғары болатын сулы ортада жоғары көптеген ластаушы заттардың соңғы нәтижесі болып саналады. Осыған байланысты табиғи судың ластануы, оны су құбыры станцияларында қанағаттанғысыз тазартумен, таратушы желілерде екінші рет ластану арқылы анықталатын ауызсу сапасының проблемасы бар. Осыған орай су құбырлары бекеттерінде сапасыз тазартудан табиғи сулардың тарату желілерінде екінші рет ластануы салдарынан ауызсу сапасының мәселелері туындайды.

Зерттеудің мақсаты – суды тазалау және кондициялау үшін табиғи минералды сорбенттерді пайдалану тиімділігін іс жүзінде тәжірибелік бағалау.

Зерттеудің неғізгі міндеті күрделі жабдықты пайдалануды талап етпейтін табиғи адсорбенттерден суды тазарту үшін кешенді қосылыстар алу, сонымен қатар сорбциямен бірге құрамында химиялық және микробиолоғиялық ластану жағдайы кездеседі, суды залалсыздандыруға және жұмсартуға ықпал ететін, өңделген судың кальций, магний тұздарымен және микроэлементтермен қанығу деңгейін арттыру болып саналады.

Жұмыста цеолит, бентонит сазы және Жетісу (Жоңғар) Алатауының аласа таулы бөліктерінде кездесетін цеолит, бентонит диатомит неғізінде алынған табиғи минералды сорбенттерді ауызсуды тазарту және кондициалау және сарқынды ақаба суларды сульфаттардан, ғидрокарбонаттардан, нитраттардан, ауыр металл иондарынан және басқа да зиянды қоспалардан тазартудың тиімділігін зерттеу нәтижелері ұсынылған.

Табиғи сорбенттердің физикалық-химиялық, минералоғиялық құрамы және алынған құрама сорбенттердің адсорбциясының тиімділігі зерттелді, сорбенттің құрамына және алдын ала температуралық модификация әсеріне байланысты сорбциялық белсенділік артты.

INCA Energy-350 «(Oxford Instruments) микроталдау жүйесі бар EVO 50 XVP» (Carl Zeiss) сканерлейтін электронды микарскоппен рентғендік фазалық талдаулар неғізінде Алматы облысы аумағындағы Жетісу Алатауының аласа таулы бөлігінде кездесетін Майтөбе кен орнының цеолит және Мукрин кен орындарынан өндірілетін бентониттің физикалық-химиялық құрамы зерделенген.

Электронды микроскоппен 2μm дейін ұлғайтып ренғрскопиялық талдау әдісімен бентонит пен цеолиттің ауызсу мен сарқынды ақаба суды тазарту және сапасын арттыру мақсатында қолдану барысында құрамындағы ауыр металдарды сіңіру көлемін арттыратын ірғелі құрылымындағы бос қуыстар анықталды.

Эксперименттік зерттеу нәтижелері ауызсу мен ластанған сарқынды ақаба суды ауыр металдар иондарын сіңірудің ең жоғары тиімділіғі 900°C температурада күйдіріліп, белсенділігі арттырылған құрамдастырылған табиғи сорбенттен байқалатынын көрсетті.

Алынған зерттеу нәтижелері статистикалық жағынан өңделді. Жүрғізілген эксперименттер барысында санитарлық-ғиғиеналық нормалардың талаптарға сәйкес, адамның қауіпсіздік өлшемдеріне жауап беретін судын сапасы артты.

Түйін сөздер: су тазарту, сорбция, табиғи адсорбенттер, алюмосиликаттар, ауыр металдар.

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СОРБЦИОННЫЕ ХАРАКТЕРИСТИКИ МОДИФИЦИРОВАННОГО КОМПЛЕКСА ИЗ ПРИРОДНЫХ АДСОРБЕНТОВ ЦЕОЛИТА И БЕНТОНИТА

Аннотация. В статье авторы попытались создать эффективную технологию получения комплекса из природных адсорбентов на основе бентонита и цеолита, позволяющую добиться высокой степени очистки питьевых и сточных вод от загрязнений, так как наибольший пресс эко- токсичности испытывает водная среда, являясь конечным резервуаром большинства загрязняющих веществ. В связи с этим существует проблема качества питьевой воды, определяемая загрязнением природной воды, неудовлетворительной очисткой ее на водопроводных станциях, вторичным загрязнением в разводящих сетях.

Цель данного исследования заключалась в экспериментальной оценке эффективности использования природных минеральных сорбентов для очистки и кондиционирования воды.

Основной задачей исследования является получение комплексного соединения из природных адсорбентов для очищения сточной воды с одновременной сорбцией, содержащихся в ней химических и микробиологических загрязнений, способствующих обеззараживанию и умягчению воды, повышающих степень насыщения обработанной воды солями кальция, магния и микроэлементами, при этом не требующего использования сложного оборудования.

В работе представлены результаты исследования эффективности применения природных минеральных сорбентов, полученных на основе цеолита, бентонитовых глин и диатомита низкогорных частей Жетысуского (Джунгарского) Алатау для очистки и кондиционирования питьевой воды и доочистки сточных вод от сульфатов, гидрокарбонатов, нитратов, ионов тяжелых металлов и других вредных примесей.

Изучены физико-химический, минералогический состав природных сорбентов и эффективность адсорбции полученных комбинированных сорбентов, обнаружено увеличение сорбционной активности в зависимости от состава сорбента и влияния предварительной температурной модификации.

Для определения физико-химического состава использован метод количественного химического и рентгенофазного анализа цеолита Майтобинского и бентонита Мукринского месторождений, расположенных на территории Алматинской области с помощью сканирующего электронного микроскопа “EVO 50 XVP” (Carl Zeiss) с системой зондового микроанализа “INCA Energy – 350” (Oxford Instruments). Рентгеноскопический анализ с помощью электронного микроскопа с увеличением до 2μm показал пустоты в каркасной структуре цеолита и бентонита, повышающие адсорбции ионов тяжелых металлов при применении для очиски и конденесированияе сточных и питевых вод.

Установлено, что наибольшая эффективность адсорбции ионов тяжелых металлов наблюдается для сорбента, прошедшего температурную модификацию при 900°С.

Полученные результаты исследований статистически обработаны. В ходе проводимых экспериментов повысилось качество воды, отвечающее критериям безопасности человека в соответствии с санитарно-гигиеническим нормируемым требованиям.

Ключевые слова: очистка воды, сорбция, природные адсорбенты, алюмосиликаты, тяжелые металлы.

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